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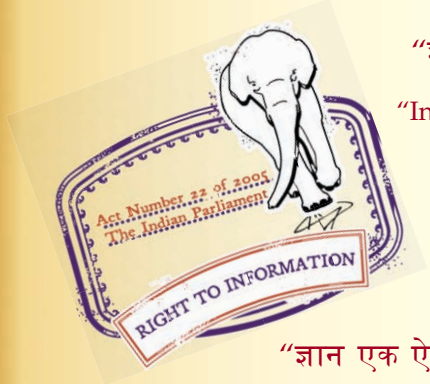
IS 5952 (1970): Methods for determination of parathion residues [FAD 1: Pesticides and Pesticides Residue Analysis]



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IS : 5952 - 1970
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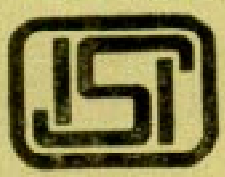
METHOD FOR DETERMINATION OF PARATHION RESIDUES

UDC 633·1 : 632·95·028 : 543·432



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NEW DELHI 1

November 1971

Indian Standard

METHOD FOR DETERMINATION OF PARATHION RESIDUES

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Indian Standard

METHOD FOR DETERMINATION OF PARATHION RESIDUES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 29 December 1970, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Due to the increased use of pesticides in the country, the question of their harmful residues and their effect on public health has been engaging the attention of the people concerned with public health. With a view to helping in collection of authentic data on pesticidal residues and in order to enable the health authorities to implement the existing Prevention of Food Adulteration Rules relating to the limits of pesticidal residues, the Indian Standards Institution has undertaken the task of preparing a series of Indian Standard methods of test for estimation of pesticidal residues.

0.3 While preparing these standards, due attention has been paid to the methods of test being followed in various laboratories of the country. For the sake of wider adoption, only those methods have been prescribed in these Indian Standards which, as far as possible, avoid the use of costly equipment and reagents.

0.4 Prior to the estimation of pesticidal residues, it is necessary to identify qualitatively the pesticidal residues. Also, it is important that the solvents that are used in these methods of test are pure and solvents are purified before analysis. Taking into consideration these two important factors, two standards covering these fields are under preparation. These standards would be necessary adjuncts to this standard.

0.5 Parathion formulations have become popular in the agricultural and public health field for the control of various pests and the method of test for estimating its residues as described in the standard is one in the series of the Indian Standard methods of test for estimating residues of various pesticides in different foodstuffs.

0.6 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the method of test for estimating parathion [*O O*-diethyl *O*-(4-nitrophenyl) phosphorothioate] foods and their milled products.

1.1.1 The method will be applicable to a minimum limit of 25 µg of parathion residues.

2. OUTLINE OF THE METHOD

2.1 The residues of parathion are extracted with benzene or *iso*propanol-benzene and the cleaned up solution is clarified. The nitro group of the parathion is then reduced with zinc dust and hydrochloric acid solution to amino group. The resulting water soluble compound is diazotized and coupled with *N*-(1-naphthyl) ethylene diamine to produce a magenta colour, the intensity of the colour is measured with a spectrophotometer at 555 to 560 mµ.

3. SAMPLING

3.1 The representative samples for the purpose of estimating parathion residues in various agricultural and food products shall be in accordance to the sampling procedures as prescribed in the relevant Indian Standards, wherever available.

4. EXPERIMENTAL PROCEDURE

4.1 Reagents

4.1.1 *Benzene* — analytical grade and redistilled.

4.1.2 *Adsorbent Mixture* — 10 parts of anhydrous powdered sodium sulphate, 5 parts of Atta clay, 5 parts Filter Cel, 2 parts of activated carbon.

4.1.3 *Parathion Standard Solution* — Prepare a standard solution for parathion in such a manner that 1 ml of the final solution contains 20 µg of parathion.

4.1.4 *Standard Hydrochloric Acid Solution* — 0·5 N.

4.1.5 *Sodium Nitrite Solution* — aqueous, 0·25 percent (*w/v*).

4.1.6 *Ammonium Sulphamate Solution* — aqueous, 2·5 percent (*w/v*). Dissolve 1·25 g in water and dilute to 50 ml. Prepare weekly.

4.1.7 *N-(1-Naphthyl) Ethylenediamine Di-Hydrochloride Solution* — aqueous, 1 percent (*w/v*). Prepare fresh solution daily and store the above in a dark coloured bottle.

4.1.8 *Zinc Dust* — analytical reagent-grade.

4.1.9 Iso*propyl Alcohol* — analytical reagent grade.

4.2 Preparation of Sample, Clean-up and Extraction of Parathion Residues

4.2.1 *Firm Relatively Tough-Skinned Fruits, such as Apples, Pears, etc* — Weigh 2 to 3 kg of fruit into a clean dry jar of 15 litres, so mounted that it can be turned with end-over-end tumbling action by hand-crank or motor. Add 500 ml of benzene (*see* Notes 1 and 2) and stopper with tight-fitting cork, wooden bung, or plastic screw-cap faced with gasket of sheet-cork or other suitable solvent-resisting material. Turn the jar for 5 minutes at 75 to 100 rpm (*see* Note 3). Open the jar and drain off benzene as completely as possible into 1-litre erlenmeyer flask (*see* Note 4).

NOTE 1 — In all cases; treat the benzene extract by shaking for five minutes with the adsorbent mixture, in proportion of approximately 10 g to 100 ml extract. Finally, filter through rapid, folded paper.

NOTE 2 — As benzene vapours are toxic, take precautions against inhaling them at all times.

NOTE 3 — Since apples treated for longer periods become swollen and soft, limit the time of stripping; on other firmer products time factor is not so important.

NOTE 4 — It is assumed that concentration of parathion in benzene poured off from apples is same as that in solvent retained by apples after wetting.

4.2.1.1 Aliquots of this solution may be used directly for parathion residue determination if they do not contain more than 100 mg of foreign organic matter.

4.2.1.2 In order to remove partially any colouring matter and plant waxes, add, to each 100 ml of the benzene solution, 10 g of a mixture compounded by weight by mixing 10 parts of anyhydrous sodium sulphate, 5 parts Atta clay, 5 parts Filter-Cel and 2 parts Nuchar. Stopper flask, shake vigorously for five minutes, and filter on folded paper (*see* Note). Do not allow appreciable evaporation of the filtrate before determining parathion in aliquot.

NOTE — This operation dehydrates the benzene, promotes rapid filtration and colouring matter and about approximately half of the natural waxes that may be present in the products.

4.2.2 *Soft Fruits, such as Plums, Tomatoes, Berries, etc* — Use 1 to 2 kg sample with 300 to 500 ml of the redistilled benzene, and strip by shaking gently by hand for five minutes in a jar of suitable size.

4.2.3 *Fresh, Leafy Vegetables, such as Cabbage, Lettuce, Greens, etc* — Blend 100 g of the sample into 100 ml of *iso*propyl alcohol for two minutes in high speed blender. Add 200 ml of benzene and blend again for two minutes,

pour the mixture into a centrifuge bottle and centrifuge for 5 minutes. Transfer supernatants to a 1-litre separator. Wash blender with 50-ml portions of benzene and transfer to centrifuge bottles. Break up solids with stirring rod, stopper and shake for 2 minutes. Centrifuge as before and add solvent layer to separator. Wash and extract with equal volume of distilled water to remove *iso*propyl alcohol. Dry benzene layer with 30 g of anhydrous sodium sulphate and dilute to suitable volume with benzene and finally make the volume to 200 ml with benzene.

4.2.4 *Other Products* — Grind dried products, in a suitable mill and extract with benzene in a large Soxhlet-type extractor for a period of one to two hours. Satisfactory extraction may be achieved in many cases by letting ground material steep overnight in stoppered jars with measured volume of redistilled benzene. Follow the same procedure as detailed in **4.2.3.**

4.3 Preparation of Standard Curve for 0-200 µg Range — Add 2·0, 4·0, 6·0, 8·0, 10·0 ml parathion standard solution B to a series of 250-ml conical flasks. Make up the volume to 25 ml with benzene in all cases and provide a blank of 25 ml benzene. Add 20 ml of dilute hydrochloric acid and 200 mg of finely powdered zinc dust and connect to a condenser by all glass-adapter fitted with thermometer. Place on a hot-plate at medium heat and rapidly distil off the benzene. Where the vapour temperature is about 70°C, ebullition stops, benzene is eliminated and vapour temperature falls. Do not let aqueous solution to boil at this point, disconnect the flask, add 10 ml of alcohol and reconnect to reflux condenser. Reflux for 5 minutes and cool the flask in ice water. Treat the other flasks likewise in succession.

4.3.1 Add about 100 mg of Filter Cel to each flask and filter into 50-ml volumetric flask through Whatman No. 44 or equivalent. Use long thin stirring rods during transfer of solutions and washing to filter. Take care not to exceed the volume of 45 ml in the flasks. Volume of wash water that may be used is limited to about 15 ml.

4.3.2 Add 1 ml of sodium nitrite solution to each flask, mix and let stand for 10 minutes, add 1 ml of ammonium sulphamate solution to each flask, mix, and let stand for 10 minutes. Then add 2·0 ml of reagent *N*-(1-naphthyl) ethylene diamine dihydrochloride solution, dilute to 50 ml volume and let stand for 10 minutes. Take the absorbance of the solution at 555 mµ in spectrophotometer (*see* Note). Colours should be stable for at least one hour.

NOTE — With 1-cm cell absorbance for 100 mcg of parathion is about 0·33. If available 5-cm cell may be preferred.

4.3.3 Read the absorbance in succession using blank standard as reference and plot optical density against concentration in micrograms of parathion to obtain standard curve.

4.4 Determination — Place aliquot of clarified solution containing the extract of parathion residues obtained as under **3.2** from the material under investigation in a 250-ml conical flask, the volume of aliquot taken depending upon the expected residue content. If the aliquot is less than 25 ml dilute to 25 ml with redistilled benzene, add 20 ml of 0·5 N hydrochloric acid and 200 mg of zinc dust and proceed as for the standard solution of parathion for the preparation of standard curve.

4.5 Reporting of Parathion Residues — Develop colour, read absorbance against standard blank and determine content of parathion in the aliquot taken from the standard curve and content of parathion residues in the material in terms of parts per million calculated from the same.

(*Continued from page* 2)

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Printed at Arcee Press, New Delhi, India